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1,2-Bis[(1,3-benzodioxol-5-yl)methylidene]hydrazine

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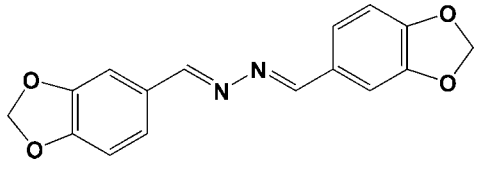
Key indicators: single-crystal X-ray study; T = 170 K; mean σ(C–C) = 0.002 Å; R factor = 0.043; wR factor = 0.118; data-to-parameter ratio = 17.5.

The complete molecule of the title compound, $C_{16}H_{12}N_2O_4$, is generated by the application of a centre of inversion. The (1,3-benzodioxol-5-yl)methylidene fused-ring system is approximately planar (r.m.s. deviation = 0.020 Å) and is essentially coplanar with the central hydrazine group [dihedral angle = 5.08 (9)°]. Weak π–π intermolecular interactions are observed [centroid–centroid distance = 3.8553 (8) Å], providing some packing stability.

Related literature

For the biological activity of Schiff bases, see: Aydogan *et al.* (2001); Desai *et al.* (2001); El-Masry *et al.* (2000); Hodnett & Dunn (1970); Pandey *et al.* (1999); Singh & Dash (1988); Taggi *et al.* (2002); Xu *et al.* (1997). For the crystallography and coordination chemistry of compounds containing the azine functionality or a diimine linkage, see: Xu *et al.* (1997); Kundu *et al.* (2005). For related structures, see: Liu *et al.* (2007); Odabaşoğlu *et al.* (2007); Zhang & Zheng (2008); Zheng *et al.* (2005*a*,*b*).



Experimental

Crystal data

$C_{16}H_{12}N_2O_4$
M_r = 296.28
Monoclinic, $P2_1/c$
a = 6.1835 (2) Å
b = 4.5970 (2) Å
c = 23.8487 (10) Å
β = 96.080 (4)°
V = 674.10 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 170 K
0.28 × 0.25 × 0.08 mm

Data collection

Oxford Diffraction Xcalibur Eos Gemini diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2010)
T_{min} = 0.971, T_{max} = 0.992
4759 measured reflections
1746 independent reflections
1402 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.118
S = 1.04
1746 reflections
100 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2010); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK5026).

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supporting information

Acta Cryst. (2012). E**68**, o7 [doi:10.1107/S1600536811050793]

1,2-Bis[(1,3-benzodioxol-5-yl)methylidene]hydrazine

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S1. Comment

Schiff bases are used as substrates in the preparation of a number of industrial and biologically active compounds via ring closure, cycloaddition and replacement reactions. Moreover, Schiff bases are also known to have biological activities such as antimicrobial (El-Masry *et al.*, 2000 & Pandey *et al.*, 1999), antifungal (Singh & Dash, 1988), antitumor (Hodnett & Dunn, 1970; Desai *et al.*, 2001), and as herbicides. Schiff bases have also been employed as ligands for complexation of metal ions (Aydogan *et al.*, 2001). On the industrial scale, they have a wide range of applications such as dyes and pigments (Taggi *et al.*, 2002). Compounds containing an azine functionality or a diimine linkage have been investigated in terms of their crystallography and coordination chemistry (Xu *et al.*, 1997; Kundu *et al.*, 2005).

The crystal structures of some Schiff base hydrazines, viz., 4-fluorobenzaldehyde [(*E*)-4-fluorobenzylidene]hydrazone (Odabaşoğlu *et al.*, 2007), *N*,*N*′-bis(3 nitrobenzylidene)hydrazine (Zheng *et al.*, 2005*a*), *N*,*N*′-bis(4-chlorobenzylidene)hydrazine (Zheng *et al.*, 2005*b*), 1,2-bis(2-chlorobenzylidene)hydrazine (Zhang & Zheng, 2008), *N*,*N*′-bis(4-hydroxybenzylidene)hydrazine (Liu *et al.*, 2007) have been reported. In view of the importance of Schiff base hydrazines, the crystal structure of title compound (I) is reported

In the crystal structure of the title compound, $C_{16}H_{12}N_2O_4$, the two 1,3-benzodioxol-5-ylmethylidene rings are planar to the hydrazine group and to each other (Fig. 1). Weak π–π intermolecular interactions are observed (centroid–centroid distance = 3.8553 (8) Å) providing some packing stability (Fig. 2).

S2. Experimental

A mixture of piperanal (3.0 g, 0.02 mol) and hydrazine hydrate (0.6 ml, 0.012 mol) was refluxed in 15 ml of absolute alcohol containing 2 drops of sulfuric acid, for about 3 hours. On cooling, the solid separated was filtered and dried. Single crystals were grown from DMF by slow evaporation. Yield: 81%. (*M*.pt.: 476 K).

S3. Refinement

All of the H atoms were placed in their calculated positions and then refined using the riding model with C—H lengths of 0.93 Å (CH) or 0.97 Å (CH_2), and with U_{iso}(H) set to 1.19-1.20 (CH, CH_2) × U_{eq}(parent atom).

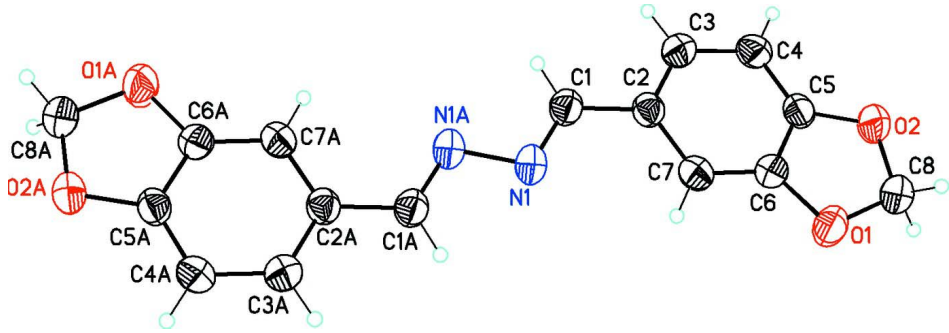

Figure 1

Molecular structure of the title compound showing the atom labeling scheme and 50% probability displacement ellipsoids.

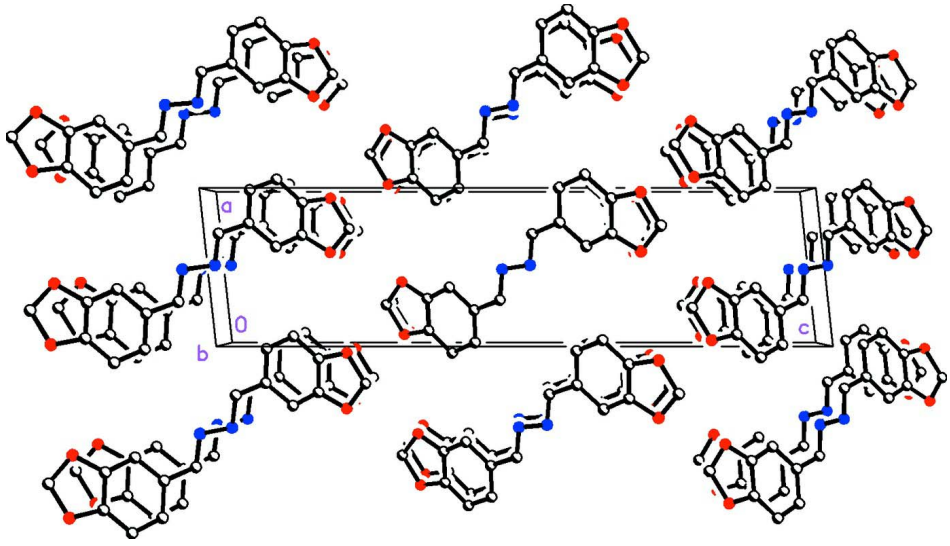

Figure 2

Packing diagram of the title compound viewed along the *b* axis. The H atoms have been removed for clarity.

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Crystal data

$C_{16}H_{12}N_2O_4$
$M_r = 296.28$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 6.1835\ (2)$ Å
$b = 4.5970\ (2)$ Å
$c = 23.8487\ (10)$ Å
$\beta = 96.080\ (4)°$
$V = 674.10\ (5)$ Å^3
$Z = 2$

$F(000) = 308$
$D_x = 1.460$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1659 reflections
$\theta = 3.4–30.1°$
$\mu = 0.11$ mm^{-1}
$T = 170$ K
Plate, yellow
0.28 × 0.25 × 0.08 mm

Data collection

Oxford Diffraction Xcalibur Eos Gemini diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.1500 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2010)
T_{min} = 0.971, T_{max} = 0.992
4759 measured reflections
1746 independent reflections
1402 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 30.1°, θ_{min} = 3.4°
h = −8→8
k = −5→6
l = −32→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.118
S = 1.04
1746 reflections
100 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0592P)^2 + 0.1301P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.40433 (16)	0.1982 (3)	0.30379 (4)	0.0593 (4)
O2	0.05345 (15)	0.0196 (2)	0.29688 (4)	0.0480 (3)
N1	0.48668 (18)	0.9022 (3)	0.47706 (4)	0.0426 (3)
C1	0.2915 (2)	0.8116 (3)	0.46809 (5)	0.0382 (3)
H1A	0.1891	0.8790	0.4909	0.046*
C2	0.22381 (19)	0.6056 (3)	0.42323 (5)	0.0347 (3)
C3	0.0129 (2)	0.4965 (3)	0.41852 (5)	0.0400 (3)
H3A	−0.0823	0.5609	0.4435	0.048*
C4	−0.0601 (2)	0.2930 (3)	0.37729 (6)	0.0417 (3)
H4A	−0.2004	0.2178	0.3747	0.050*
C5	0.0850 (2)	0.2105 (3)	0.34108 (5)	0.0358 (3)
C6	0.2948 (2)	0.3188 (3)	0.34523 (5)	0.0367 (3)
C7	0.36960 (19)	0.5151 (3)	0.38540 (5)	0.0385 (3)
H7A	0.5111	0.5862	0.3877	0.046*
C8	0.2520 (2)	0.0174 (4)	0.27125 (6)	0.0469 (4)
H8A	0.3079	−0.1794	0.2701	0.056*

H8B	0.2271	0.0900	0.2329	0.056*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0448 (6)	0.0813 (9)	0.0538 (6)	−0.0115 (5)	0.0141 (5)	−0.0309 (6)
O2	0.0443 (5)	0.0526 (7)	0.0463 (6)	−0.0050 (4)	0.0008 (4)	−0.0174 (5)
N1	0.0472 (6)	0.0415 (7)	0.0384 (6)	−0.0023 (5)	0.0016 (5)	−0.0110 (5)
C1	0.0428 (7)	0.0355 (7)	0.0360 (6)	0.0014 (5)	0.0027 (5)	−0.0033 (5)
C2	0.0375 (6)	0.0323 (7)	0.0335 (6)	0.0014 (5)	−0.0005 (5)	−0.0003 (5)
C3	0.0372 (6)	0.0429 (8)	0.0406 (7)	0.0009 (5)	0.0067 (5)	−0.0035 (6)
C4	0.0321 (6)	0.0450 (8)	0.0476 (7)	−0.0035 (5)	0.0023 (5)	−0.0043 (6)
C5	0.0370 (6)	0.0340 (7)	0.0347 (6)	0.0006 (5)	−0.0036 (5)	−0.0020 (5)
C6	0.0356 (6)	0.0405 (7)	0.0342 (6)	0.0014 (5)	0.0039 (5)	−0.0020 (5)
C7	0.0327 (6)	0.0421 (8)	0.0402 (7)	−0.0036 (5)	0.0015 (5)	−0.0031 (5)
C8	0.0476 (8)	0.0507 (9)	0.0420 (7)	0.0016 (6)	0.0026 (6)	−0.0110 (6)

Geometric parameters (Å, º)

O1—C6	1.3728 (15)	C3—C4	1.3970 (19)
O1—C8	1.4228 (17)	C3—H3A	0.9300
O2—C5	1.3697 (15)	C4—C5	1.3630 (18)
O2—C8	1.4284 (18)	C4—H4A	0.9300
N1—C1	1.2732 (17)	C5—C6	1.3836 (18)
N1—N1^{i}	1.413 (2)	C6—C7	1.3607 (18)
C1—C2	1.4566 (18)	C7—H7A	0.9300
C1—H1A	0.9300	C8—H8A	0.9700
C2—C3	1.3907 (18)	C8—H8B	0.9700
C2—C7	1.4043 (18)		
C6—O1—C8	106.27 (10)	C4—C5—O2	128.05 (12)
C5—O2—C8	105.97 (10)	C4—C5—C6	121.98 (12)
C1—N1—N1^{i}	111.66 (14)	O2—C5—C6	109.97 (11)
N1—C1—C2	121.97 (12)	C7—C6—O1	128.17 (12)
N1—C1—H1A	119.0	C7—C6—C5	122.35 (12)
C2—C1—H1A	119.0	O1—C6—C5	109.48 (11)
C3—C2—C7	120.11 (12)	C6—C7—C2	117.09 (11)
C3—C2—C1	119.19 (12)	C6—C7—H7A	121.5
C7—C2—C1	120.69 (12)	C2—C7—H7A	121.5
C2—C3—C4	121.90 (12)	O1—C8—O2	108.17 (11)
C2—C3—H3A	119.1	O1—C8—H8A	110.1
C4—C3—H3A	119.1	O2—C8—H8A	110.1
C5—C4—C3	116.56 (12)	O1—C8—H8B	110.1
C5—C4—H4A	121.7	O2—C8—H8B	110.1
C3—C4—H4A	121.7	H8A—C8—H8B	108.4
N1^{i}—N1—C1—C2	−179.47 (14)	C8—O1—C6—C5	2.58 (16)
N1—C1—C2—C3	174.71 (13)	C4—C5—C6—C7	−0.3 (2)

N1—C1—C2—C7	−4.6 (2)	O2—C5—C6—C7	179.55 (12)
C7—C2—C3—C4	1.0 (2)	C4—C5—C6—O1	179.77 (13)
C1—C2—C3—C4	−178.40 (13)	O2—C5—C6—O1	−0.38 (16)
C2—C3—C4—C5	−1.2 (2)	O1—C6—C7—C2	179.93 (13)
C3—C4—C5—O2	−178.96 (13)	C5—C6—C7—C2	0.0 (2)
C3—C4—C5—C6	0.9 (2)	C3—C2—C7—C6	−0.3 (2)
C8—O2—C5—C4	177.86 (14)	C1—C2—C7—C6	179.01 (12)
C8—O2—C5—C6	−1.98 (15)	C6—O1—C8—O2	−3.78 (16)
C8—O1—C6—C7	−177.34 (15)	C5—O2—C8—O1	3.55 (16)

Symmetry code: (i) −*x*+1, −*y*+2, −*z*+1.